Color, True and Apparent, LR

Platinum-Cobalt Standard Method^{1, 2, 3}

3 to 200 color units

Scope and application: For water, wastewater and seawater; equivalent to NCASI method 253 and NCASI Method Color 71.01 for pulp and paper effluent using 465 nm (pH adjustment necessary).

- ¹ Adapted from Standard Methods for the Examination of Water and Wastewater and National Council for Air and Stream Improvement (NCASI) Methods Manual.
- ² Adapted from Wat. Res. Vol. 30, No. 11, pp. 2771-2775, 1996.
- ³ NCASI Method 253 approved at 40 CFR part 136.

☐ Test preparation

Instrument-specific information

Table 1 shows all of the instruments that have the program for this test. The table also shows sample cell and adapter requirements for this test. To use the table, select an instrument, then read across to find the applicable information for this test.

Table 1 Instrument-specific information

| Instrument | Adapter | Sample cell |
|------------------------------------|-----------------|-------------|
| DR 3900, DR 3800, DR 2800, DR 2700 | — | 2629250 |
| DR 5000 | A23618 | |
| DR 6000 | LZV902.99.00020 | |

Before starting

The NCASI procedure is available only for spectrophotometers and pH adjustment to pH 7.6 with 1.0 N HCl or 1.0 N NaOH is a requirement. If the volume change during the adjustment is more than 1%, start again and use a stronger acid or base. Use Program 124 for the NCASI procedure.

To minimize the volume change from the dilution, add one pH 8 buffer powder pillow (sodium phosphate/potassium phosphate) to 50 mL of sample before the final pH adjustment . Fully mix to dissolve before the final pH adjustment.

To test for apparent color, do not filter the sample or the deionized water blank.

Refer to Method technique on page 4 for precautions on low-level color measurements.

Review the Safety Data Sheets (MSDS/SDS) for the chemicals that are used. Use the recommended personal protective equipment.

Dispose of reacted solutions according to local, state and federal regulations. Refer to the Safety Data Sheets for disposal information for unused reagents. Refer to the environmental, health and safety staff for your facility and/or local regulatory agencies for further disposal information.

Items to collect

| Description | Quantity |
|--|----------|
| Buffer, pH 8.0 (Program 124) | 1 |
| Hydrochloric Acid Solution, 1.0 N (Program 124) | varies |
| Sodium Hydroxide, 1.00 N (Program 124) | varies |
| Water, deionized | 100 mL |
| Filter apparatus: membrane filter, filter holder, filter flask and aspirator | 1 |

Items to collect (continued)

| Description | Quantity |
|---|----------|
| Stopper, rubber, one hole, number 7 | 1 |
| Tubing, rubber | 1 |
| Sample cells (For information about sample cells, adapters or light shields, refer to Instrument-specific information on page 1.) | 2 |

Refer to Consumables and replacement items on page 6 for order information.

Sample collection and storage

- Collect samples in clean glass or plastic bottles.
- Analyze the samples as soon as possible for best results.
- If prompt analysis is not possible, fill the bottle completely full, then tighten the cap on the bottle. Avoid excessive agitation or prolonged contact with air.
- To preserve samples for later analysis, keep the samples at or below 6 °C (43 °F) for up to 48 hours.
- Let the sample temperature increase to room temperature before analysis.

Platinum-Cobalt Standard Procedure





1. Start program 121 LR Color, 455 nm. For the NCASI test, program 124 LR Color, 465 nm. For information about sample cells, adapters or light shields, refer to Instrumentspecific information on page 1.

Note: Although the program name can be different between instruments, the program number does not change.

2. Collect 200 mL of sample in a 400-mL beaker. NCASI: Adjust the pH as described in Before starting on page 1.

3. Assemble the 0.45 micron filter apparatus. NCASI: Use a 0.8-micron filter for the NCASI test. For samples that are difficult to filter, use a 1.0 micron

prefilter first.



4. Pour approximately 50 mL of deionized water through the filter. Discard the filtered water.



5. Pour another 50 mL of deionized water through the filter.



6. Prepare the blank: Fill the sample cell with 10 mL of filtered deionized water from the previous step. Discard the remaining water in the flask.



7. Clean the blank sample cell.



Insert the blank into the cell holder.
DR 2700, DR 2800, DR
3800 and DR 3900: Slide the sample cell to the right side of the cell compartment to consistently align the cell. Close the lid.



9. Push **ZERO**. The display shows 0 units PtCo 50 mm.



10. Pour approximately 50 mL of sample through the filter.



11. Prepare the sample: Fill a second sample cell with 10 mL of filtered sample.



12. Clean the prepared sample cell.



13. Insert the prepared sample cell into the cell holder.

DR 2700, DR 2800, DR 3800 and DR 3900: Slide the sample cell to the right side of the cell compartment to consistently align the cell. Close the lid.

Accuracy check

Standard solution method

Use the standard solution method to validate the test procedure, the reagents and the instrument.

14. Push READ. Results

Read

14. Push **READ**. Results show in units PtCo 50 mm.

Items to collect:

- Standard solution within the test range
- 1. Use the test procedure to measure the concentration of the standard solution.
- 2. Compare the expected result to the actual result.

Note: The factory calibration can be adjusted slightly with the standard adjust option so that the instrument shows the expected value of the standard solution. The adjusted calibration is then used for all test results. This adjustment can increase the test accuracy when there are slight variations in the reagents or instruments.

Method performance

The method performance data that follows was derived from laboratory tests that were measured on a spectrophotometer during ideal test conditions. Users can get different results under different test conditions.

| Program | Standard | Precision (95% Confidence Interval) | Sensitivity Concentration change per 0.010 Abs change |
|--------------|----------------|-------------------------------------|--|
| 121 (455 nm) | 15 units Pt-Co | 14 to 16 units Pt-Co | 7.4 units Pt-Co |
| 124 (465 nm) | 15 units Pt-Co | 14 to 16 units Pt-Co | 7.6 units Pt-Co |

Method technique

Zero and read the sample

Use good laboratory technique and give attention to detail for accurate, reproducible results on waters that have true color values less than 15 Pt-Co color units.

- 1. Clean the sample cell with soap and water or with an acid-rinse to remove contaminants (e.g., dirt, grease, finger prints and so on).
- 2. Rinse the sample cell several times with filtered deionized water.
- 3. Fill the cleaned sample cell with 6 mL of deionized water.
- 4. Use a lint-free towel to clean the sample cell windows.
- 5. Make sure that there are no trapped air bubbles and no lint or smudges on or in the sample cell.
- 6. Carefully tap the sample cell to remove trapped air bubbles.
- 7. Insert the sample cell into the cell holder. Then push **Zero**. Note: The sample cell may initially fit tightly in the sample cell holder, but will loosen slightly with additional use. There may be some small variations between cell manufacturers.
- 8. Remove the sample cell and do steps 1 and 2 again to clean the cell.
- 9. Fill the cleaned sample cell with deionized water. Do steps 4 to 6 again.
- **10.** Put the sample cell in the holder, then push **Read**.

If the instrument does not show "0", the sample cell was not correctly cleaned. Make sure that there are no trapped bubbles in the sample cell and the sample cell is correctly aligned.

Filter the sample cell

It is not known if apparent color values are meaningful in low level color measurements. Turbidity or suspended particles can contribute to the measured color value and can cause high results. It is recommended that all samples for low level color measurements are first filtered through a membrane filter and the results are reported as true color.

- 1. Set the instrument to Zero.
- **2.** Discard the deionized water and rinse the sample cell at least two times with the filtered sample.
- **3.** Fill the sample cell with filtered sample. Make sure that there are no bubbles or smudges on the sample cell.
- 4. Put the cell into the cell holder to do a measurement.

Clean the sample cell

After the measurements are completed, fully rinse the sample cell with deionized water, then dry the sample cell. Put the sample cell in an environment with no dust. Make sure that this environment is only for LR color measurements. For more sample measurements do the cleaning procedure again.

Summary of method

Color may be expressed as apparent or true color. Apparent color includes color from dissolved materials and color from suspended matter. True color is determined by removal of the suspended materials with a filter or a centrifuge. This method uses 0.45-micron filtration for true color analysis. To measure apparent color, do not filter the sample or the deionized water blank. The same instrument program is used for both true and apparent color. The stored program is calibrated in color units based on the APHA-recommended standard of 1 color unit being equal to 1 mg/L platinum as chloroplatinate ion. Test results for Programs 121 and 124 are measured at 455 and 465 nm, respectively in spectrophotometers. Test results for Program 122 are measured at 420 nm in colorimeters.

Set up the instrument

The steps that follow are general instructions for all instruments. Refer to the instrument documentation that is used for the correct menu options.

For instruments that do not have programs 121 and 124, make a new user program as follows.

- 1. Set the instrument power to on, then let the instrument complete the self-check.
- **2.** Obey the prompts on the display to enter commands for the 455 nm or 465 nm methods.
- 3. Go to User Programs.
- 4. Select a new user program and enter the settings for each option:

| Option | Description |
|----------------------------------|--|
| Program Number (950–999)? | Select a Program Number |
| Program Name? | Enter the program name: LR_Color_455_nm or LR_Color_465_nm |
| Program Type | Select Single Wavelength |
| Units | Enter the applicable units |
| Wavelength (nm) | Select 455 or 465 nm |
| Concentration resolution | Select the concentration resolution: 1 |
| Chemical form? | Select the chemical form: PtCo_50_mm |
| Calibration | Enter Formula |
| Enter formula | 455 nm: b=737.70 465 nm: b=764.00 |
| User Progam for number assigned | Upper Limit |
| Upper limit | ON>220 |
| User Program for number assigned | Lower Limit |
| Lower limit | ON>3 |

5. Make the necessary selection to save, edit or exit an option.

Consumables and replacement items

Required reagents

| Description | Quantity/test | Unit | ltem no. |
|-----------------------------------|---------------|--------|----------|
| Water, deionized | varies | 4 L | 27256 |
| Required reagents (Program 124): | | | |
| Buffer, pH 8.0 | 1 | 15/pkg | 1407995 |
| Hydrochloric Acid Solution, 1.0 N | varies | 1 L | 2321353 |
| Sodium Hydroxide, 1.0 N | varies | 1 L | 104553 |

Required apparatus

| Description | Quantity/test | Unit | ltem no. |
|---|---------------|---------|----------|
| Aspirator, vacuum pump | 1 | each | 213100 |
| Beaker, 400-mL | 1 | each | 50048 |
| Filter holder, 47-mm, magnetic base | 1 | each | 1352900 |
| Filter, membrane, 47-mm, 0.8-microns, Program 124 | 1 | 100/pkg | 2640800 |
| Filter, membrane, 47-mm, 0.45-microns | 1 | 100/pkg | 1353000 |
| Flask, filtering, 500-mL | 1 | each | 54649 |
| Sample cell, rectangular glass, 5-cm | 1 | each | 2629250 |
| Stopper, poly, hollow | 1 | 6/pkg | 211907 |
| Tubing, rubber, 7.9 mm x 2.4 mm | varies | 12 ft | 56019 |

Optional reagents and apparatus

| Description | Unit | ltem no. |
|--|---------|----------|
| Color Standard Solution, 500 platinum-cobalt units | 1 L | 141453 |
| Color Standard Solution, 15 platinum-cobalt units | 1 L | 2602853 |
| Filter, glass microfiber, 1.0-micron 47-mm | 100/pkg | 2551400 |
| Flask, volumetric, Class A, 500-mL glass | each | 1457449 |
| Hydrochloric Acid Solution, 6.0 N (1:1) | 500 mL | 88449 |
| Liqui-Nox Phosphate-free detergent | 946 mL | 2088153 |
| Pipet, volumetric, Class A, 50-mL | each | 1451541 |
| Pipet filler, safety bulb | each | 1465100 |
| Sample cell, 5-cm rectangular quartz | each | 2624450 |
| Wipes, disposable | 280/pkg | 2097000 |

